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On the ductility of alpha titanium: The effect of temperature and deformation mode

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Abstract

Single phase α-titanium shows anomalous warm deformation behaviour. As the temperature increases, ductility increases in uniaxial tension and decreases in biaxial stretching. Previously, this behaviour was attributed to an increase in strain rate sensitivity and a decrease in twinning activity with temperature. In this study, we show that it can instead be explained by an increase in slip anisotropy with temperature. Grade 2 CP-Ti sheet was tested in uniaxial tension at 20 °C and 300 °C to determine ductility, work hardening behaviour and the coefficient of plastic anisotropy (R-value). The increase in uniaxial ductility with temperature was found to be a consequence of an increasing rate of saturation work hardening with temperature.
In the absence of significant twinning, this unexpected work hardening behaviour was attributed to an increase in slip anisotropy with temperature. This hypothesis was supported by crystal plasticity finite element modelling results, which are also able to predict the observed increase in surface roughness with temperature. The increase in anisotropy leads to higher strain localization which, coupled with the increasing work hardening rate helps explain why biaxial ductility decreases with increasing temperature. In addition to explaining the limitations in warm forming of Ti, understanding the origins of these effects contributes to our general understanding of the deformation of other hexagonal metals like Zr and Mg.

**Keywords:** Titanium; work hardening; anisotropy; formability; crystal plasticity finite element modelling

1.- Introduction

Despite its hexagonal crystal structure and consequent lack of easy slip systems, α-titanium has excellent ductility and formability at room temperature [1–5]. Nevertheless, commercial purity titanium (CP-Ti) is not usually cold formed, primarily because of the strong rate of work hardening, which contributes to the setting up of high residual stress and high levels of spring-back. The amount of spring-back could potentially be reduced significantly via warm forming. In CP-Ti the main source of strengthening is interstitial solid solution strengthening, primarily by oxygen, and therefore its flow stress depends strongly on temperature: the flow stress of CP-Ti grade 2 (containing max. 0.25 wt. % of oxygen) is some 3 times lower at 300 °C than at room temperature. Although this lower flow stress produces much less spring-back, CP-Ti sheet is usually formed above 500 °C, near the recrystallization
temperature [6]. This need for hot, rather than warm forming, arises in part because
titanium has poor biaxial ductility and bendability when formed warm [7,8].
However, this decrease in formability with increasing temperature is only manifested
during biaxial stretching. In uniaxial tension, the ductility increases with increasing
temperature, as expected [1,9].

This apparently contradictory behaviour is not well known and has not been
studied in detail, possibly because the forming of CP-Ti sheet is currently of only
limited technological importance. However this behaviour is reported and discussed
in Ti conference articles from the 1980's, primarily by Itoh et al [7] and Roberts and
Wilson [8], where the increase in ductility in uniaxial tension with increasing
temperature is attributed to an increase in strain rate sensitivity, whereas the decrease
in biaxial ductility with increasing temperature is attributed to “a decrease in through
thickness ductility” caused by increased surface roughness with increasing
temperature. However, these explanations fail to explain why the ductility of Ti
depends on the deformation mode, since the strain rate sensitivity in CP-Ti is
dominated by dislocation solute interactions [10,11], which should be unaffected by
the change in deformation mode and the associated change in slip activity.
Furthermore, the increase in surface roughness with temperature is seen after both
uniaxial and biaxial stretching. Why does it only affect ductility in the biaxial case?

In this article, we report the results of a detailed study on the effect of
temperature and loading mode on the ductility of grade 2 CP-Ti. The aim was to
understand the origin of this anomalous behaviour. We tested the material in uniaxial
and biaxial tension, reproducing the anomalous behaviour reported previously and
used electron-backscatter diffraction to characterise the material and measure the
texture changes caused by deformation. The results of these tests revealed that the
increase in ductility in uniaxial tension with temperature is due to a difference in work hardening behaviour that is unrelated to twinning. We used crystal plasticity finite element modelling to show that this work hardening behaviour could be caused by an increase in single crystal plastic anisotropy with temperature, which also leads to the observed increase in surface roughness.

2.- Experimental procedure

2.1- Material

The material studied is grade 2 commercially pure titanium (CP-Ti) ($\leq 0.07$ wt. % Fe, $\leq 0.08$ wt. % C, $\leq 0.03$ wt. % N, $\leq 0.25$ wt. % O, $\leq 60$ ppm H, bal. Ti) in the form of 1 mm thickness rolled sheet.

2.2- Microstructural characterization

We used EBSD (Electron Backscattered Diffraction) to characterize the microstructure with a FEI Sirion equipped with an Oxford Instruments EBSD camera operating at an acceleration voltage of 20 kV and using a step size of 0.25 µm. Samples were ground to #4000 grit paper and finished with OP-S (0.2 µm) suspension for ~30 min. EBSD data was analysed using the commercial Channel 5 software (Oxford Instruments). The contour pole figures were plotted using $10^\circ$ data clustering. In the EBSD maps after deformation, the twinned region was identified by first highlighting the twin boundaries in Channel 5 and then filling in the area contained by these twin boundaries. The twinned area fraction was subsequently obtained using the image analysis software ImageJ [12] by measuring the area fraction.

2.3- Mechanical characterization
Dog-bone tensile samples with 60 x 12.5 x 1 mm gauge dimensions were electro-discharge machined along the rolling direction (RD) and the traverse direction (TD). The samples were tested in an Instron 5589 universal testing machine equipped with a furnace at $\dot{\varepsilon} = 10^{-4}$ s$^{-1}$ and at room temperature (RT) and 300° C. The strain was measured using a clip-on extensometer (gauge length of 50 mm) and using digital image correlation (DIC). DIC was used primarily for measuring the strains along the width but also give strain along the length. Measurements of longitudinal strain using the extensometer showed excellent agreement with those from DIC and either could have been used. However, because the DIC measurements show more scatter at small strains, the extensometer values were preferred in the production of the stress strain ($\sigma$-$\varepsilon$) curves. Besides the true stress-strain curves obtained from the tensile tests, the work hardening ($\Theta$) behaviour was studied by plotting the relationship $\Theta = \partial \sigma / \partial \varepsilon$ versus the true strain ($\varepsilon$) for each testing condition. We also evaluated the strain rate sensitivity ($m$) using Eq. 1 for different tensile tests at different strain rates in the range $\dot{\varepsilon} = 10^{-4} – 10^{-2}$ s$^{-1}$.

$$m = \left[ \frac{\sigma}{\varepsilon} \right]$$  \hspace{1cm} [1]

To assess the change in formability during biaxial deformation, a set of Erichsen tests was carried out using a heated blank and punch at RT, 100° C and 300° C in an Erichsen 145 testing machine at a punch speed of 60 mm/min, using discs 13 mm in diameter and 1 mm thick. This punch speed produces strain rates in the biaxial stretch test to those used during uniaxial testing. Finally, we measured the surface roughness after mechanical testing using a confocal Bruker Contour Elite K with profiler function over a 170×130 µm area.
2.4- Digital Image Correlation

We used digital image correlation (DIC) to measure strain along the tensile sample’s width ($\varepsilon_w$) and length ($\varepsilon_l$) at RT and 300° C. A pattern was applied onto the sample surface using Dupli-Color Aqua ® high quality lackspray (white speckles), followed by Dupli-Color Acryl Deco Matt ® (black speckles) which provide a good variation in contrast, needed to perform the DIC calculations. We used a LaVision Imager ProX 4 Megapixel ® (2048x2048 pixel resolution) camera to track the displacement of the paint speckles. The camera consists of a 14-bit charged couple device chip which was Peltier cooled to -15°C in order to reduce the background noise, using an image acquisition frequency of 10 Hz. The recorded sets of images were processed using LaVision Davis 8.2 ® correlation software using a final interrogation window of 32 x 32 pixels with a 25% overlap. We used Numerical Python [13] routines and representations using Matplotlib [14] to analyse the DIC data.

The displacement map at each frame was differentiated using a using second order accurate central differences method as implemented in Numpy [13]. The DIC analysis produces full-field in-plane displacement maps $u(x_1, x_2, 0)$ on the plane $x_1x_2$ with normal $x_3$. The instant total strains along the direction $i$ are calculated as:

$$\varepsilon_i = \ln \left( 1 + \frac{\partial u_i}{\partial x_i} \right) \quad [2]$$

In this study, the directions $i$ are denoted as $w$, $t$ and $l$ and correspond to the sample width, thickness and length, respectively. We used the values of $\varepsilon_w$ and $\varepsilon_l$ to calculate the plastic strain ratio (R-ratio), a measure of the plastic anisotropy of a sheet metal.

Assuming volume constancy, $\Delta V/V = \varepsilon_l + \varepsilon_w + \varepsilon_t = 0$, the R-ratio is defined as follows:

$$R\text{-ratio} = \frac{\varepsilon_w}{\varepsilon_t} = \frac{\varepsilon_w}{-\varepsilon_t - \varepsilon_w} \quad [3]$$
Therefore we can use the DIC measurements of $\varepsilon_w$ and $\varepsilon_l$ to calculate a total R-ratio which includes both elastic and plastic strains. This is different from the R-ratio calculated from the final shape or plastic strain alone.

2.5- Crystal Plasticity Finite Element Modelling

The effect of the critical resolved shear stresses (CRSS) variations when testing at the two studied temperatures was simulated using three-dimensional crystal plasticity finite element modelling (CPFEM). The model consisted in a mesh of 15x15x15 20-node quadratic brick-shaped elements containing 8 integration points (IPs) per element. Orientations were given per element; hence each of the 8 IPs of an element had the same orientation, giving a total of 3375 grains. In the case of the modelling of the biaxial deformation mode we used a 32x32x3 arrangement of elements, representing 3072 grains distributed in a sheet with three grains across the thickness.

A rate sensitive formulation was used in which plastic deformation is assumed to occur via slip according to:

$$\frac{\dot{\gamma}^\alpha}{\dot{\gamma}_0} = \left( \frac{\tau^\alpha}{\tau_{\text{CRSS}}} \right)^{\frac{1}{m}}$$

[4]

where $\dot{\gamma}^\alpha$ is the shear strain rate in the $\alpha$ slip system, $\dot{\gamma}_0$ is a nominal reference slip rate, $\tau^\alpha$ is the resolved shear stress in the $\alpha$ slip system, $\tau_{\text{CRSS}}^\alpha$ is the critical resolved shear stress in the $\alpha$ slip system and $m$ is the rate sensitivity ($m=0.02$). The evolution of the CRSS values is governed by

$$\Delta \tau_{\text{CRSS}}^\alpha = \sum_{\beta=1}^{n} H_{\alpha\beta} |\Delta \gamma^\beta|$$

[5]

where $\Delta \gamma^\beta$ is the shear strain increment and $H_{\alpha\beta}$ is the hardening matrix between two slip systems $\alpha$ and $\beta$. 
\[ H_{\alpha\beta} = L_{\alpha\beta} \cdot \theta^\alpha; \quad L_{\alpha\beta} = \begin{pmatrix} 1 & \cdots & 1 \\ \vdots & \ddots & \vdots \\ 1 & \cdots & 1 \end{pmatrix} \]  

\[ L_{\alpha\beta} \] is a matrix of 1s, indicating that all the slip systems interact between each other with the same strength. Finally, the hardening rate for the \( \alpha \) slip system, \( \theta^\alpha \), can be expressed using the Voce hardening equation:

\[ \theta^\alpha = \theta_{IV} + \theta_1 \left( 1 - \frac{\tau^\alpha}{\tau_s} \right)^\alpha \]  

where \( \theta_{IV} \) is the work hardening rate in the stage IV, \( \theta_1 \) is the initial work hardening rate at \( \sigma = 0 \), \( \tau_s \) is the saturation shear stress and \( \alpha \) controls the rate of change in hardening with shear stress. In this case we considered 4 slip families, namely: (a) basal, (b) prismatic \(<a>\), (c) pyramidal \(<a>\) and (d) pyramidal \(<c+a>\). In the present model, we intentionally selected the hardening parameters to fit just the initial curvature in the tensile curves, at low strain values. The initial slip resistances and hardening parameters are listed later in the discussion of the modelling results. A detailed description on the solution algorithm used can be found in [15].

3.- Results

3.1- Microstructural characterization

The EBSD map in the RD–TD plane and pole figures of the as-received material are showed in Fig. 1. The microstructure is made up of equiaxed grains with an average size of about 20 \( \mu m \). However, there is some variation of grain size in the material, with some regions containing grains up to 100 \( \mu m \) in diameter. The \{0001\} pole figure presents a predominance of basal planes parallel to the RD–TD plane but split around ND towards TD. This is a typical texture for this material in the rolled and recrystallized condition which produces mechanical anisotropy.
Fig. 2a and b show EBSD maps on the RD-ND plane for samples after tensile testing to an elongation of 15% at $\dot{\varepsilon} = 10^{-4} \text{ s}^{-1}$ at RT and 300 °C, respectively. Although there is a noticeable difference in the grain size of the grains in the two maps, this is simply a result of variability in the starting material and not a consequence of dynamic recrystallization or grain growth. CP-Ti does not recrystallize at 300 °C, as the grain misorientation evident in the EBSD map confirms.

The maps show orientation gradients within each grain which is evidence of substructure formation. Additionally, Fig. 2c and d highlight the twins extracted from each map in Fig. 2a and b at RT and 300 °C, respectively. The twined material volume fraction is around 2.5 % at RT and 1 % at 300 °C, and is made up of both compression $\{11\overline{2}\}\{11\overline{2}\overline{3}\}$ and tension $\{10\overline{1}\overline{2}\}\{10\overline{1}\overline{1}\}$ twins at RT while only a few tension $\{10\overline{1}\overline{2}\}\{10\overline{1}\overline{1}\}$ twins are observed at 300 °C. Finally, Fig. 2e and f present the pole figures obtained from the maps in Fig. 2a and b, respectively. As expected, the microtexture after deformation remains essentially similar to the as-received material.

### 3.2- Mechanical characterization

The true $\sigma$–$\varepsilon$ tensile curves at a strain rate of $10^{-4} \text{ s}^{-1}$ are shown in Fig. 3a, for the two testing directions RD and TD, at RT and 300 °C. The material has a noticeable upper yield point, which has been attributed to interstitial oxygen [1]. The yield stress ($\sigma_{0.2}$) and the maximum flow stress ($\sigma_{\text{max}}$) decrease sharply with the modest increase in testing temperature for both testing directions. For instance, the $\sigma_{0.2}$ values at RT are 288 MPa for RD and 316 MPa for TD whereas at 300 °C values are 72 MPa and 74 MPa respectively. The $\sigma_{\text{max}}$ values at RT are 504 MPa for RD and 465 MPa for TD and 200 MPa and 170 MPa at 300 °C respectively. The ductility increases with increasing testing temperature for both testing directions. The values of
elongation to failure, $e_F$ are 43% for RD and 38% for TD at RT while at 300 °C it reaches values of 59 and 57% for RD and TD respectively. Comparing the mechanical behaviour between the two testing directions, in general, the values of $\sigma_{0.2}$ when testing along RD are lower than TD. Nevertheless, RD has higher values of $\sigma_{\text{max}}$. Additionally, the ductility is higher when testing along RD than TD. This difference in behaviour with testing direction is probably a consequence of the crystallographic texture of the rolled sheet (Fig. 1).

The evolution of the work-hardening rate with strain is shown in Fig. 3b. At the onset of plastic deformation, the curves exhibit a hump followed by a smooth decrease. Previous work has attributed this behaviour to the occurrence of twinning after yielding, especially at room temperature [16–18]. However, there are only a few twins in the EBSD maps in Fig. 2, suggesting that there is only a very limited amount of twinning, in accordance with other more recent work [1]. Instead this brief work hardening rate increase at low deformation values is likely to be related to the upper yield point observed, which is probably caused by interstitial oxygen [1,9]. As expected, the work hardening at low strains is higher at RT than at 300 °C. At RT, the work hardening rate starts to drop just after a strain of 0.17 and 0.26 for the TD and RD directions respectively, which does not happen at 300°C until the strain reaches around 0.35. The necking onset obtained from the Considere’s criteria is highlighted with a black dot in both, the true $\sigma$–$\varepsilon$ tensile curves and the work-hardening rate curves.

A consequence of this unusual hardening behaviour is that the uniform elongation, $\varepsilon_u$, is higher at 300 °C than at RT. At the same time, the elongation after necking ($\Delta\varepsilon_p$) is only slightly higher at 300 °C than at RT (Fig. 4), which suggests there is only a small increase in strain rate sensitivity with increasing temperature.
This implies that the increase in ductility with increasing temperature is almost entirely due to an increase in $\varepsilon_u$, represented by $\Delta \dot{\varepsilon}_T$ in Fig. 4. This observation is consistent with the strain rate sensitivity ($m$) values measured at different strain rates ($10^{-4}$–$10^{-3}$ s$^{-1}$), which for both testing directions are around 0.03 at RT and 0.04 at 300 °C, in agreement with other measurements [19]. The strain rate sensitivity changes gradually with temperature and strain, suggesting the same deformation mechanisms are operative in the temperature and strain regimes tested.

To assess the plastic anisotropy of the material at different temperatures, the R-ratio was calculated from DIC strain data obtained during the test (Fig. 3c). An R-ratio higher than one indicates that, during plastic deformation, $\varepsilon_t$ the strain in the thickness direction (ND) is lower than the strain along the width $\varepsilon_w$ (RD, TD). Given the starting texture of the material, with alignment of the 0002 poles around ND, an R-ratio higher than one is expected. The measured R-ratio, shown in Fig 3c, lies between 3 and 4 for all tests after a strain of 0.05 where it is essentially constant. The highest value is for the tests along TD, for which it is temperature independent. When loading along RD, the R-ratio at 300° C is higher than that at RT, suggesting anisotropy increases with temperature. There is a peak in R-ratio at yield in all tests, which is higher for TD loading and which broadens with increasing temperature. However, its significance cannot be easily determined, since the R-ratio definition used here contain elastic strain contributions.

The biaxial stretching tests confirmed the previous finding [7,8] that, unlike in uniaxial tension, ductility during biaxial deformation decreases with increasing temperature. Fig. 5 shows the drawing force ($F$) vs. punch stroke ($d$) curves at three testing temperatures. The curves reveal that, as expected from the tensile test results,
the required force for deforming the material is lower at higher testing temperatures than at room temperature. However, whereas in tension the ductility increased with temperature, here the maximum punch stroke decreases. More precisely, the maximum dome height at 300 °C is two-thirds of that at room temperature.

4.- Discussion

The mechanical testing results confirm that CP-Ti behaves atypically in the temperature range studied. There is an anomalous increase in saturation work hardening and anisotropy with increasing temperature in uniaxial tests and whereas the ductility decreases with increasing temperature in biaxial stretching, it increases in uniaxial stretching.

4.1- Anomalous higher work hardening behaviour at high testing temperature

The main source of strain hardening is usually assumed to be the interaction between dislocations [20–23]. Therefore, strain hardening should decrease with increasing temperature, since recovery will happen more readily at high temperatures [24,25]. Therefore, it is unsurprising that the initial work hardening rate is higher at RT than at 300º C. However, with increasing plastic strain, the work hardening rate at RT saturates and drops just before necking occurs. At 300º C, on the other hand, this drop with strain is much more gradual suggesting a different source of work hardening exists at this temperature. This is an important observation since it is this higher hardening rate, which coupled with the lower flow stress, delays necking and increases ductility.

Our EBSD observations exclude twinning as a possible source of this enhanced hardening and support the idea that there should be less twinning at higher
temperatures [1,26]. If twin activity is negligible, then the increased plastic anisotropy, as measured by the R-ratio, must be due to changes in relative slip activity.

Although the slip resistance of hard slip modes like $<c+a>$ slip decreases with increasing temperature, this is true for all slip systems. However, making hard slip systems easier does not necessarily decrease slip anisotropy. The anisotropy is not determined by the $<c+a>$ slip resistance but instead by the ratio of $<a>$ and $<c+a>$ slip resistance. If the thermal component of the slip resistance is an additional term and similar in magnitude for both slip systems, then increasing temperature can increase the ratio of slip resistances, even if both decrease.

This idea is consistent with the variation of measured critical resolved shear stress (CRSS) values for $<a>$ and $<c+a>$ with temperature reported in Lutjering and Williams for Ti–6.6Al alloy [26]. Although the slip resistance decreases for all slip systems, the CRSS($<c+a>$)/CRSS($<a>$) ratio actually increases with increasing temperature [27]. CP-Ti can be even more anisotropic than its alloys [5,28] and therefore, this effect is possibly even more pronounced than in the Al containing alloy.

The consequence of this increasing slip anisotropy, is that the grains poorly aligned for easy slip become even harder to deform with increasing temperature, effectively behaving as a hard second phase, at least along certain directions. The presence of these hard grains could contribute to hardening via the generation of back-stress caused by the incompatibility in deformation between them and other better aligned, softer grains. This hardening mechanism was originally studied by Ashby [29], Brown and Stobbs [30] and Mory and Tanaka [31] in particle reinforced aluminium, where the hard phase is undeformable. It is not clear, therefore, whether
these back-stress arguments apply to CP-Ti with high single crystal plastic anisotropy. High plastic anisotropy can have pronounced effects of the early work hardening of magnesium, which can be reproduced by elasto-plastic self-consistent (EPSC) modelling [32]. However, its significance at larger strains is more difficult to assess since the predictions of EPSC models are restricted to small deformations. Nevertheless, it offers a possible explanation for the anomalous behaviour observed. To test this hypothesis, we performed a series of simulations using crystal plasticity finite element modelling (CPFEM), which should be able to reproduce the effects of increasing anisotropy on work hardening.

We used a temperature independent formulation and simulated the mechanical behaviour of CP-Ti at the two temperatures by varying the CRSS(< c + a >/CRSS(< a >)) ratios, as shown in Table 1, which also includes the compliance values, the initial CRSS values for each slip family as well as the hardening parameters at room temperature and 300°C for the prismatic slip family. Although the elastic compliance changes with temperature, this effect is small between RT and 300 °C and was ignored, as was twinning, the extent of which EBSD showed to be minimal. The texture measured using EBSD and shown in Fig. 1 was used to produce discrete representative orientations to be used in the CPFE simulations, which corresponds to the pole figures presented in Fig. 6a.

The main aim of the simulations was to explore the effect of changing anisotropy on the saturation work hardening rate. However, the initial hardening is also different at the two temperatures, since recovery occurs more readily at 300 °C than at room temperature. To account for this effect, the parameters of a Voce-type hardening law were determined by fitting the initial portion of the stress strain curves.
To keep the number of parameters to a minimum, work hardening was assumed to act
only in the prismatic slip system. The resultant change in work hardening for
prismatic slip calculated using Eq. 7 is shown in Fig. 7. The figure shows how the
hardening parameters in Table 1 only determine the initial work hardening rate and
that this contribution to hardening disappears well before necking starts. Assuming
the applied stress, $\sigma$, is related to the resolved shear stress $\tau$ by $\sigma = 2\tau$, the Voce
hardening contribution of Eq. 7 in the overall hardening finish at $\sigma = 80$ MPa and
$\sigma = 320$ MPa for the 300 °C and RT simulations respectively. This is well below
necking in both cases, as Fig. 3a shows.

The experimental and simulated uniaxial true stress – strain curves are
compared in in Fig. 8a. The curves show that a reasonable calibration is possible
using a simple hardening model for prismatic slip only. As expected, the simulated
stress-strain curves do not show an upper yield point, since there is no mechanism for
it in the crystal plasticity model. Apart from that, however, the predicted work
hardening responses, shown in Fig. 8b, capture all the main features of the
experimental work hardening curves. In particular, the model reproduces the increase
in saturation work hardening rate with increasing anisotropy at 300 °C, as well as the
increased uniform elongation it enables. There is also good agreement between the
CPFEM simulated R-ratio values, shown in in Fig. 8c, and those determined
experimentally (Fig. 3c), further supporting the idea that the plastic anisotropy
increases as the temperature increases from 300 °C to RT.

Additionally, the simulated microtexture after 15 % deformation at RT and
300 °C are presented in Fig. 6b and c, respectively. They both present good agreement
with the pole figures observed for the experimental results in Fig 2e-f, supporting the model formulation.

These CPFEM results demonstrate that increasing slip anisotropy can indeed contribute to work hardening. Although it is possible that this effect is a consequence of geometric hardening caused by texture evolution, a comparison between the predictions of a visco-plastic self-consistent model (VPSC) and CPFEM [33] has shown that the effect of texture evolution is very weak and actually produces softening in uniaxial tension. The same study also showed that the effect of anisotropy on hardening is only predicted by the CPFEM and not by the VPSC model, implying that it probably arises from the elastic interactions between grains and the resultant back-stresses, which are not accounted for in the viscoplastic self-consistent formulation. Although this effect is most noticeable at higher temperatures, anisotropy induced back-stresses must contribute to work hardening at all temperatures, including RT. Therefore, in hexagonal materials, the work hardening rate is determined by both back-stresses and dislocation hardening. The evidence here is that, in CP-Ti, the dislocation density contribution saturates very early and that the back-stress term dominates as the material approaches necking. This large contribution of back-stresses to work hardening could help explain why spring-back in CP-Ti is so pronounced and notoriously difficult to model.

At higher temperatures, the plastic anisotropy increases but the slip resistance decreases. The consequence is a delay in the onset of necking by Considére’s criterion and therefore increased ductility. Although hexagonal materials are often said to be brittle because of their high plastic anisotropy, a consequence of the limited availability of slip systems, here CP-Ti becomes more ductile with increasing
temperature because of increasing anisotropy and its effect on work hardening. The
effect on biaxial ductility, however, is the opposite.

4.2- Decreasing ductility in biaxial conditions at high testing temperature

Although increasing the temperature improves uniaxial ductility, it decreases
biaxial ductility. Our results are consistent with those of Itoh et al. [7] who showed
that the bendability of CP-Ti decreases at 300 °C before increases again at around 500
°C, closer to the usual forming temperature for CP-Ti [26]. In their study, they also
observed that by weakening the texture, i.e. decreasing the R-ratio, the formability at
300 °C can be improved. Similar results were obtained by Roberts and Wilson [8],
who found that formability deteriorates from room temperature to 200 °C before
improving again up to 500 °C. They also showed that decreasing formability when
increasing testing temperature was correlated to an increase in the surface roughness,
and that the rate at which surface roughening develops during stretching is
proportional to grain size.

Roberts and Wilson attributed the increase in surface roughening at higher
temperatures to the loss of twinning as an operative deformation mechanism, although
they presented limited evidence for it in the form of optical micrographs (EBSD had
not yet been developed). Their argument was that twinning effectively reduces the
effective grain size during deformation, reducing surface roughness [8]. However, we
found only limited amounts of twinning in the material after stretching and then
mostly near the tool and not in the bulk. This is consistent with the texture of the
material and the anisotropic nature of twinning. However, one would expect for
surface roughness to increase with increasing slip anisotropy – even in the absence of
twinning.
Our measurements of surface roughness, summarised in Table 2 and illustrated in the case of the biaxial stretching after fracture at RT and 300 °C in Fig. 9, confirm Roberts and Wilson’s correlation between decreasing biaxial ductility with increasing surface roughness with increasing temperature. In order to see if this could be a consequence of the increasing single crystal slip anisotropy, the same CPFEM model used to simulate uniaxial deformation was used to simulate biaxial stretching and the accompanying surface roughening. The results of these simulations are shown in Fig 10. The model predicts that the surface roughness increases with temperature, in agreement with the measurements. This shows that the increasing roughness can be explained by the higher \( \text{CRSS}(<c+a>)/\text{CRSS}(<a>) \) ratio at high temperatures alone and that twinning is not required to explain it. The overestimation of surface roughness in the simulation is probably a direct consequence of using only 3 grains across the thickness of the modelled volume due to computational cost, when the sheet tested contains approximately 50 grains across the thickness. The fewer the grains across the thickness, the bigger the effect of the slip anisotropy on the surface roughness. This is consistent with the correlation between starting grain size and surface roughness measured for different materials [8].

Like in the uniaxial simulations, where necking occurred when the Considéré’s criterion was met, the biaxial simulations also produce a strain localization events. Because in uniaxial testing the material is unconstrained in the transverse direction, the material can plastically deform without developing high triaxial stresses until necking occurs. In biaxial stretching however, the material deformation is much more constrained, which in turn enhances the effect of strain localization on failure by promoting the early development of triaxial stresses and
damage. Therefore, whereas in uniaxial tension the increasing slip anisotropy with temperature promotes ductility by increasing the work hardening rate, in biaxial stretching it leads to enhanced strain localisation and stress triaxiality leading to earlier failure. In other words, in tension the incompatibility between soft and hard grains increases ductility, in biaxial stretching it decreases it.

5.- Conclusions
The effect of temperature on the ductility of CP-Ti deformed warm (below the recrystallization temperature) depends on the deformation mode. Whereas increasing the temperature improves ductility in uniaxial tension it decreases it in biaxial stretching. Previously, these effects had been explained by higher strain rate sensitivity and lower twinning activity respectively. However, we have shown that they can be both explained by increasing slip anisotropy with temperature. The increasing anisotropy contributes to work hardening, extending uniform elongation in uniaxial tension and increasing ductility, whilst promoting strain localization and damage formation during biaxial stretching and decreasing ductility. The effect of plastic anisotropy on work hardening is often ignored but, as this work shows, it can have a significant effect on the behaviour of hexagonal materials. In addition to its effects on warm formability, it is probably very relevant to spring-back in these materials, which is notoriously difficult to predict.

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References

[9] M. Doner, H. Conrad, Deformation mechanisms in commercial Ti (0.5 at. pct oineq) at intermediate and high temperatures (0.3 - 0.6 tinm), Metall. Trans. 4 (1973) 2809–2817. doi:10.1007/BF02644581.


Figure captions

Fig. 1. EBSD map and pole figures obtained in the RD–TD plane for the as-received CP-Ti.

Fig. 2. EBSD map in the RD-ND plane after a macroscopic deformation of 15% at $\dot{\varepsilon}=10^{-4}$ s$^{-1}$ and at a) RT and b) 300 °C. The compression $\{11\bar{2}2\}\{11\bar{2}3\}$ and tension $\{10\bar{1}2\}\{10\bar{1}1\}$ twins are highlighted in blue and red, respectively, for the tested material at c) RT and d) 300 °C. Pole figures at e) RT and f) 300 °C after deformation.

Fig. 3. a) True stress-true strain ($\sigma$–$\varepsilon$) curves b) work hardening vs. true strain ($\Theta$–$\varepsilon$) and c) R-ratio vs. true strain (R-ratio–$\varepsilon$) at $\dot{\varepsilon}=10^{-4}$ s$^{-1}$ for the two temperatures along RD and TD. The necking onset obtained from the Considere´s criteria are highlighted with a black dot for each condition.

Fig. 4. Values of uniform elongation ($\varepsilon_u$) and elongation to failure ($\varepsilon_l$) at $\dot{\varepsilon}=10^{-4}$ s$^{-1}$ for the two temperatures along RD and TD obtained from the Considere´s criteria. The difference $\varepsilon_l - \varepsilon_u$ is represented by $\Delta\varepsilon_p$, which represents the post-uniform elongation while $\Delta\varepsilon_T$ represent the increase in elongation attributed to the increasing temperature.

Fig. 5. Drawing force vs. punch stroke (F–d) at three testing temperatures obtained from the Erichsen test.

Fig. 6. a) Initial texture used in the CPFE simulations. Pole figures obtained from the simulations after 15% deformation at b) RT and c) 300 °C.

Fig. 7. Hardening ($\Theta$) as a function of the shear stress ($\tau$) for prismatic slip at two temperatures.

Fig. 8. (a) Modelled (solid lines) and experimental (dashed lines) true stress-true strain ($\sigma$–$\varepsilon$) curves (b) modelled work hardening vs. true strain ($\Theta$–$\varepsilon$) and (e) modelled R-ratio vs. true strain (R-ratio–$\varepsilon$) for the two simulated testing temperatures when testing along RD and TD.

Fig. 9. Experimental values of the distance from the mean height under biaxial testing conditions after fracture (a) at RT and (b) at 300 °C.
Fig. 10. Roughness CPFEM simulated for both equivalent temperatures: (a) Ra–ε evolution and (b) values of the distance from the mean height in the surface at three different strain values.
Table 1. Selected compliance values, initial $\tau_{\text{CRSS}}$ and hardening parameters for the prismatic slip family used in the CFEM simulation.

<table>
<thead>
<tr>
<th>Compliance values ($\times 10^{-6}$ MPa) [31]</th>
<th>$S_{11}$</th>
<th>$S_{33}$</th>
<th>$S_{44}$</th>
<th>$S_{12}$</th>
<th>$S_{13}$</th>
<th>$S_{66}$</th>
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</thead>
<tbody>
<tr>
<td>$S_{11}$</td>
<td>9.62</td>
<td>8.01</td>
<td>29.94</td>
<td>-4.09</td>
<td>-2.46</td>
<td>28.58</td>
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<table>
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<tr>
<th>Initial critical resolved shear stress values (MPa)</th>
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<tr>
<td>RT [32]</td>
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<tr>
<td>Basal Prism. &lt;a&gt; Pyram. &lt;a&gt; Pyram. &lt;c+a&gt;</td>
</tr>
<tr>
<td>$162.4$</td>
</tr>
<tr>
<td>$20.0$</td>
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<table>
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<tr>
<th>Hardening parameters for prismatic slip family</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\theta_{IV}$ $\theta_1$ $\tau_s$ $a$</td>
</tr>
<tr>
<td>RT</td>
</tr>
<tr>
<td>$0$</td>
</tr>
<tr>
<td>$300 \degree C$</td>
</tr>
<tr>
<td>$0$</td>
</tr>
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</table>

Table 2. Roughness values ($R_a$) measured after testing at different temperatures in uniaxial and biaxial conditions. The as-received material roughness was 0.8.

<table>
<thead>
<tr>
<th>Uniaxial test</th>
<th>$T$ ($\degree C$)</th>
<th>$R_a$ ($\mu m$)</th>
<th>RT</th>
<th>300</th>
<th>500</th>
</tr>
</thead>
<tbody>
<tr>
<td>$R_a$ ($\mu m$)</td>
<td>2.919</td>
<td>3.54</td>
<td>5.461</td>
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<table>
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<tr>
<th>Biaxial test</th>
<th>$T$ ($\degree C$)</th>
<th>$R_a$ ($\mu m$)</th>
<th>RT</th>
<th>100</th>
<th>200</th>
<th>300</th>
</tr>
</thead>
<tbody>
<tr>
<td>$R_a$ ($\mu m$)</td>
<td>1.515</td>
<td>2.617</td>
<td>3.275</td>
<td>3.702</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
a) For the stress-strain curve

b) For the strain energy curve

c) For the R-ratio curve

- RT: Room Temperature
- 300 °C: 300 °C
- RD: Rolling Direction
- TD: Transverse Direction

CPFEM: Computational Plasticity Finite Element Method

Experimental: Experimental data